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14. ABSTRACT
The Aerospace Corporation has developed a space qualification method for silicon carbide optical systems that covers material verification through system development. Recent laboratory results for testing of materials properties of silicon carbide are described. These include mechanical testing and non-destructive techniques to measure elastic properties. Results of tests on samples that were part of the MISSE-6 flight experiment are included.

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Results from Mechanical Testing of SiC for Space Applications:
Non-Destructive Evaluation Samples and MISSE-6 Experiment Samples

David B. Witkin
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Mirror Technology Days 2010
Boulder, Colorado
7 June 2010

Space Qualification Goals

- Develop process and procedures for characterizing materials for future space systems applications including SiC
- Space qualify vendors for optical and structural applications
- Emphasize not only material characterization but also component and system evaluation
- 2010 Mirror Technology Days presentation:
  - *Mechanical testing results on SiC materials previously characterized by non-destructive evaluation (NDE) techniques*
  - *Results of MISSE-6 SiC mechanical testing*

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Future Space Application Challenges

- SiC is not space-qualified: Vendors may claim flight heritage equals space qualification but this is not true
- Currently, no standard test plan exists for space qualification: Aerospace is developing method
- Multiple vendors and various processes to make SiC
  - *Confidence in one vendor's product does not translate to other vendors*
  - *Understand vendor's process control and batch-to-batch variability*
- To be successful, space qualification will require support from multiple programs and agencies
- SiC database will maintain knowledge continuity during funding fluctuations
  - *Aerospace developed initial prototype: contains test data, photographs, and documents*
  - *Goal is to have virtual laboratory to aid collaboration*

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Space Qualification Method

- Four evolutionary phases

R&D

Acquisition

- **Material and Substrate Characterization**
  - Understand material strength and survivability
  - Includes mechanical tests, radiation exposure, NDE, and modeling
- **Component and Sub-system Characterization**
  - Includes thermal tests, additional mechanical tests, coating and polishing tests
  - Leads to space qualification of vendor's material
- **System Characterization**
  - Testing of telescope and assembly
    - Thermal vacuum, vibration tests, optical performance
  - Leads to space qualification of specific system
- **Post-Acquisition**
  - Post-storage system analysis, if applicable
  - Analysis of on-orbit performance throughout mission
  - End-of-life assessment of material performance
  - Provide feedback that is incorporated into R & D

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Mechanical Testing: Purpose

- Mechanical testing is necessary to characterize a structural material
  - *Mechanical properties are essential for designing structures and components subjected to mechanical loads*
- Strength of brittle materials must be assessed statistically: large number of tests must be performed
  - *Strength of brittle materials scales inversely with size of sample under stress*
  - *Large data sets improve probabilistic modeling of strength and safety factors*
- Limited space on MISSE for mechanical test samples means that results must be assessed from a different perspective

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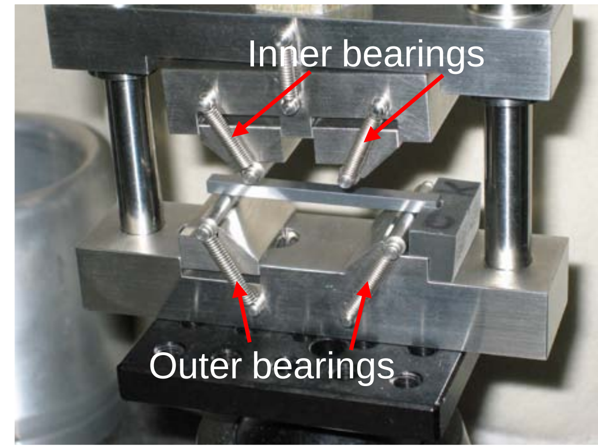
Mechanical Testing Overview

- Three different tests performed in compliance with ASTM standards:
  - *Modulus of Rupture (ASTM C 1161-02c)*
  - *Fracture Toughness (ASTM C 1421-01b)*
  - *Equibiaxial Flexural Strength (ASTM C 1499-04)*
- Test sequence includes:
  - *Measurement of specimen dimensions*
  - *Verification that specimen fracture location and test outcome are valid within ASTM standard*
  - *Photography and archiving of test specimens after testing*
- Materials from four vendors have been tested to various degrees
  - *One vendor's material was tested before and after radiation exposure*

B Bars



Disk

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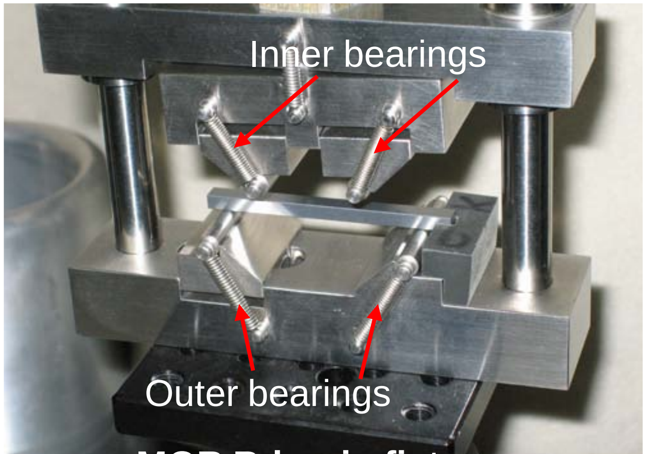
Modulus of Rupture (MOR)

Justification

Flexural strength test more reliable measure of uniaxial strength than tensile test

Four point bend testing for MOR gives uniform tensile stress in load span

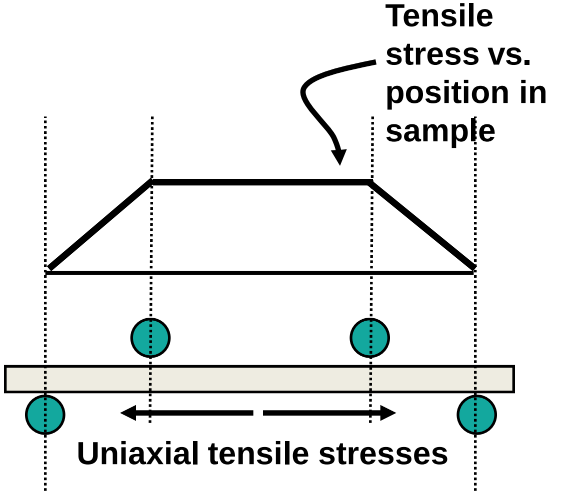
B Bars



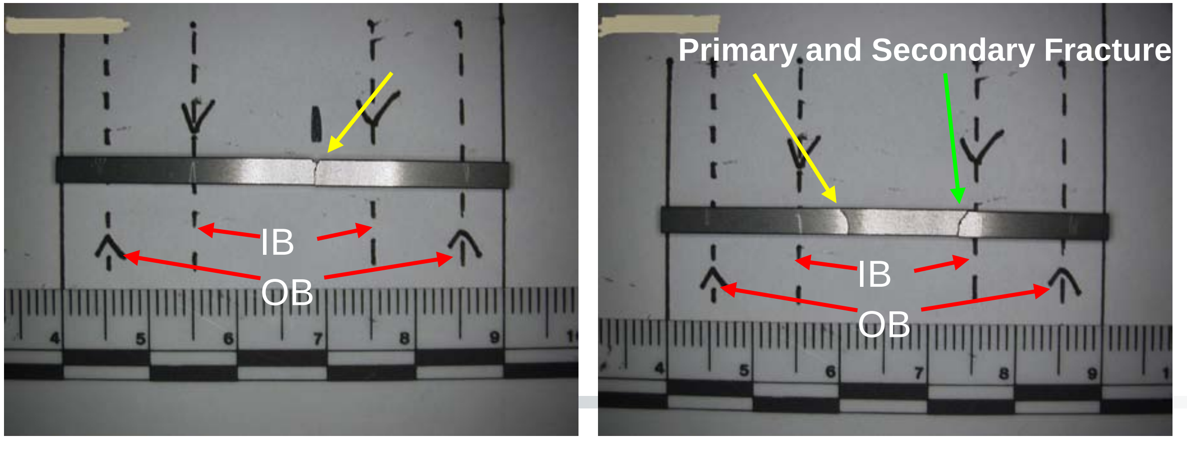
MOR B-bar in fixture

Testing Sequence

1. **Perform mechanical test; inspect fracture locations for test validity**
2. **Measure dimensions of broken B-Bar**
3. **Photograph broken sample**
4. **Intermittent surface and chamfer inspection**



Crack locations for two MOR samples



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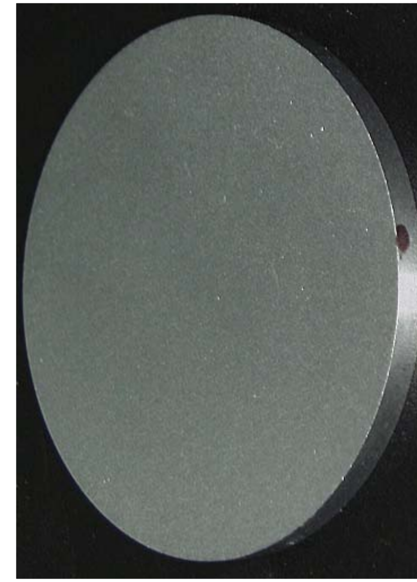
Equibiaxial Flexural Strength (EFS)

Justification

Equibiaxial flexural strength provides lowest flexural strength of a material

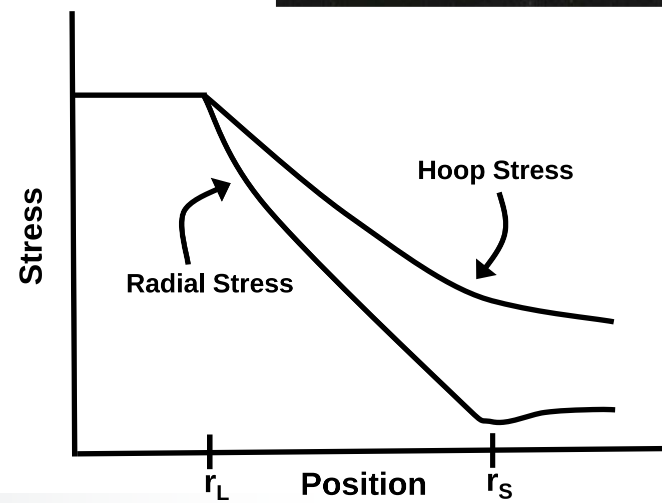
Data free of edge effects. Sample edge is at lower stress than area under load ring

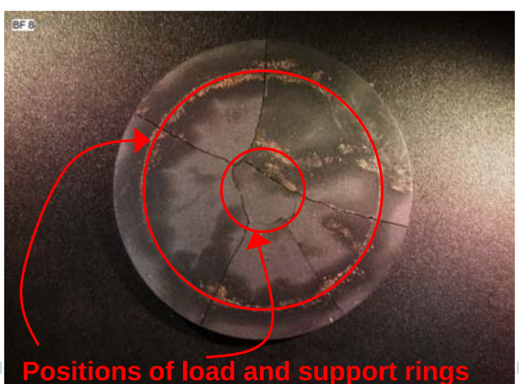
Effect of surface polishing on strength of mirror can be evaluated

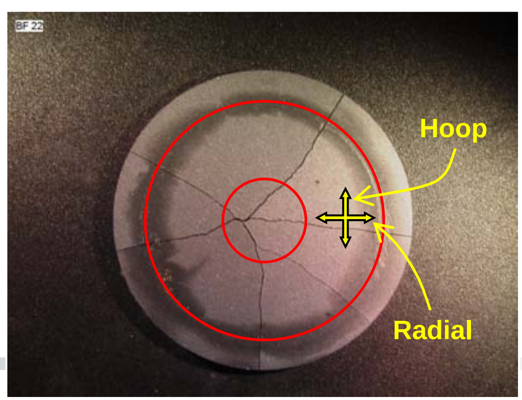


Testing Sequence

1. **Measure sample dimensions (disk diameter and thickness)**
2. **Perform test; evaluate fracture pattern for test validity**
3. **Photograph broken sample**







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Goals of Data Analysis

- Derive material properties that can be used to model component reliability at the highest levels
  - *Strength data*
    - 30 samples minimum to determine Weibull parameters
    - 30 samples minimum to determine B basis strength (90% reliability with 95% confidence)
    - 100 samples minimum to determine A basis strength (99% reliability with 95% confidence)
  - *Fracture Toughness*
    - ASTM 1421 C requires 4 valid tests per material or testing condition

- Comparing and combining test data
  - *Are different lots or batches the same?- production consistency*
  - *Are different billets from the same lot the same?- process variability*
  - *Does radiation exposure affect strength?*

- Considerations for NDE materials
  - *Do variations in material properties determined by NDE correlate with variations in strength in mechanical testing?*

- Considerations for MISSE experiment
  - *Does MISSE environment affect strength?*
    - Degradation of mechanical and optical properties of SiC after radiation exposure has been reported

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NDE Materials and Methods

For full details: “NDE methods for determining the materials properties of silicon carbide plates”, S. Kenderian et al., 2009 SPIE Proceedings, vol. 7425

- Materials
  - *10” x 16” SiC plates provided by two vendors*
    - Thickness was approximately 0.375” and 0.25” vendors 1 and 2 respectively, but non-uniform
- NDE Methods
  - *Ultrasonic C-scans*
    - Plates were immersed in water and scanned with a 25MHz ultrasonic transducer
    - Scans were made along length and width with measurements made at 0.025-inch increments
    - Reflected signal was gated three ways to isolate contributions from front surface echo, back surface echo and plate internal volume
  - *Eddy current testing*
    - Both sides of plate tested in air using 1.68 MHz AC driving current

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Calculation of Material Properties

- Acoustic measurements
  - *Measured variable is time to receive reflected longitudinal and shear waves, which is used to calculate Poisson' ratio and Young's modulus:*

$$\nu = \frac{\left(\frac{t_L}{t_S}\right)^2 - 2}{\left(\frac{t_L}{t_S}\right)^2 - 1}$$

$$E = \rho\left(\frac{t_L}{2 \cdot thickness}\right)^2 \frac{(1-2\nu)(1+\nu)}{(1-\nu)}$$

- Eddy current measurements
  - *Measured variables are resistance (R) and inductance (L) in the presence of air or the SiC plate, which are used to calculate impedance magnitude change*

$$|\Delta Z| = \left|\left(R_{plate} - R_{air}\right) + i\omega\left(L_{plate} - L_{air}\right)\right|$$

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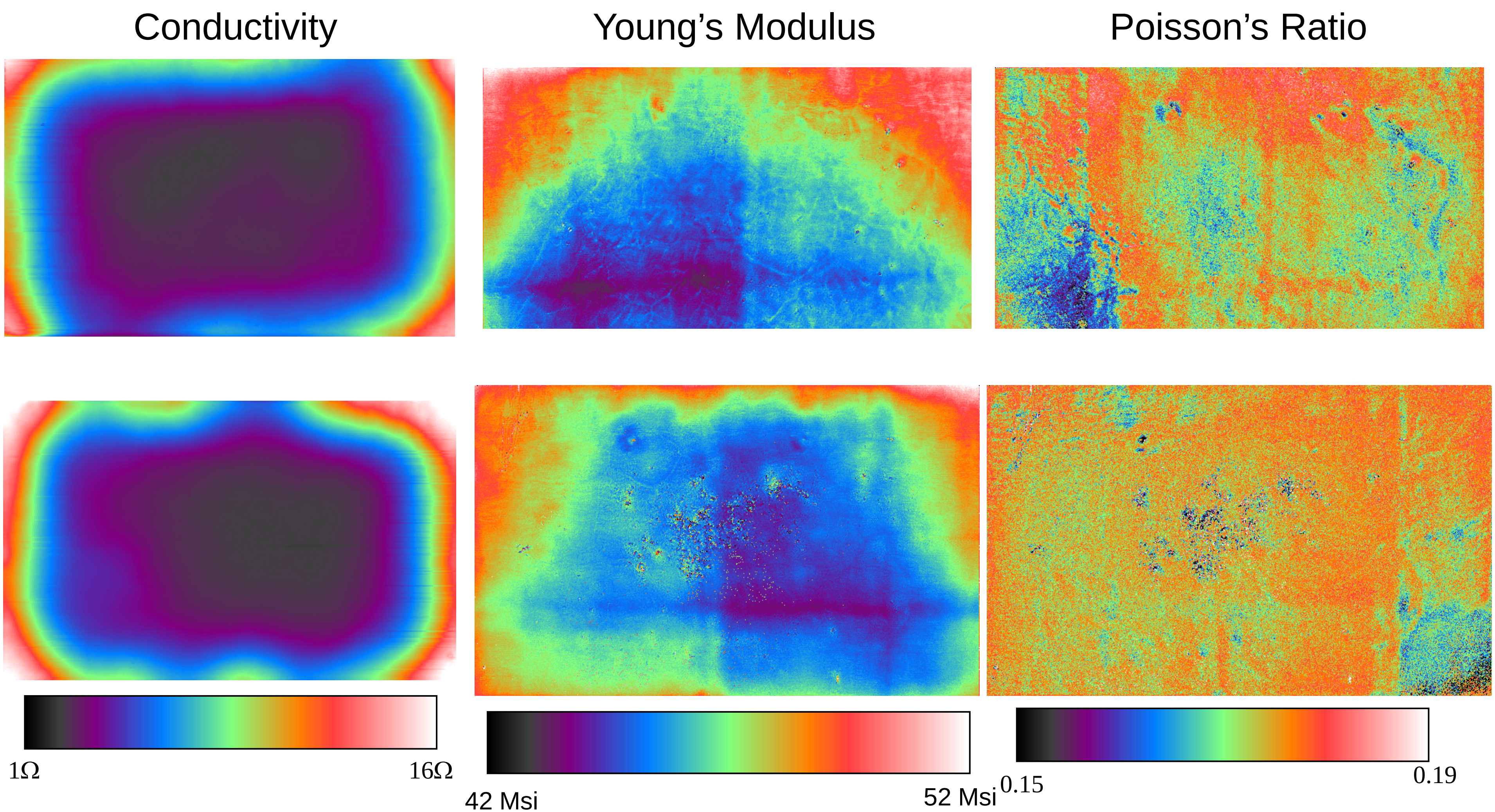
Vendor 1 Material Properties

Plate 1 top, Plate 2 bottom



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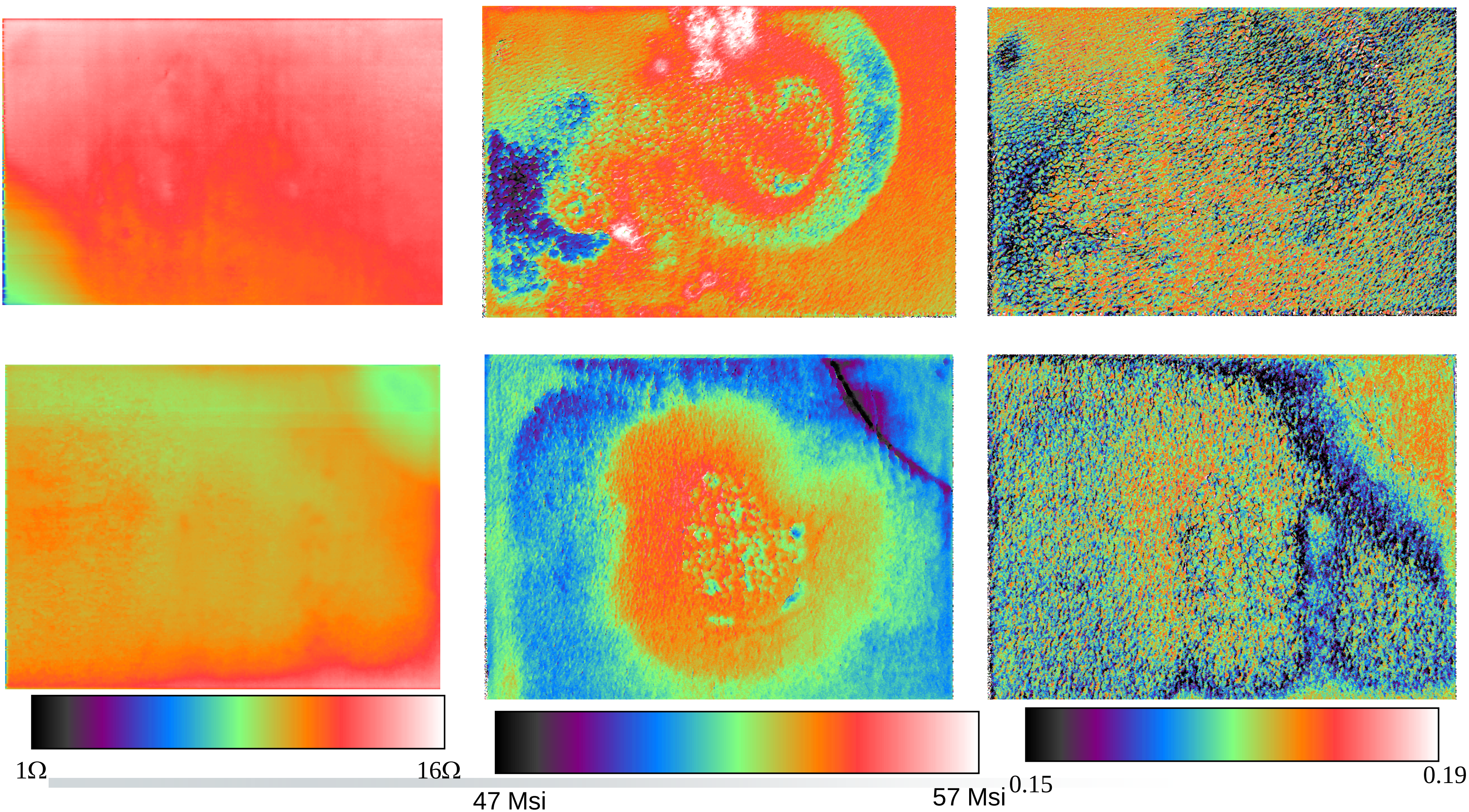
Vendor 2 Material Properties

Plate 1 top, Plate 2 bottom

Conductivity | Young's Modulus | Poisson's Ratio



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Comments on NDE Results

- Poisson's ratio *v* is calculated based on TOF only and independent of material thickness
  - *Biaxial flexural stress is related to Poisson's ratio*
- Young's modulus *E* is calculated based on velocity of longitudinal wave, which assumes constant thickness of plate and measured point-by-point Poisson's ratio
  - *Variations in E reflect both property changes AND small variations in thickness*
  - *Deflection under biaxial flexural stress is related to Young's modulus*

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Notes on Presentation of SiC Test Data

- The Aerospace Corporation is treating all vendors' test data as proprietary
- The test data on the following several slides are all real data sets, HOWEVER…
  - *Vendor names will not be used*
  - *No references to type of SiC will be given*
  - *No actual strength values will be given*
- Data belong to our government customers
  - *They may choose to share our results and/or analysis with the vendor, or direct us to do the same*
- Strength data of brittle materials are often plotted using the Weibull distribution:

$$F(\sigma) = 1 - \exp\left[-\left(\frac{\sigma}{\sigma_\theta}\right)^m\right]$$

$$\ln(-\ln(1-F)) = m\ln\sigma - m\ln\sigma_\theta$$

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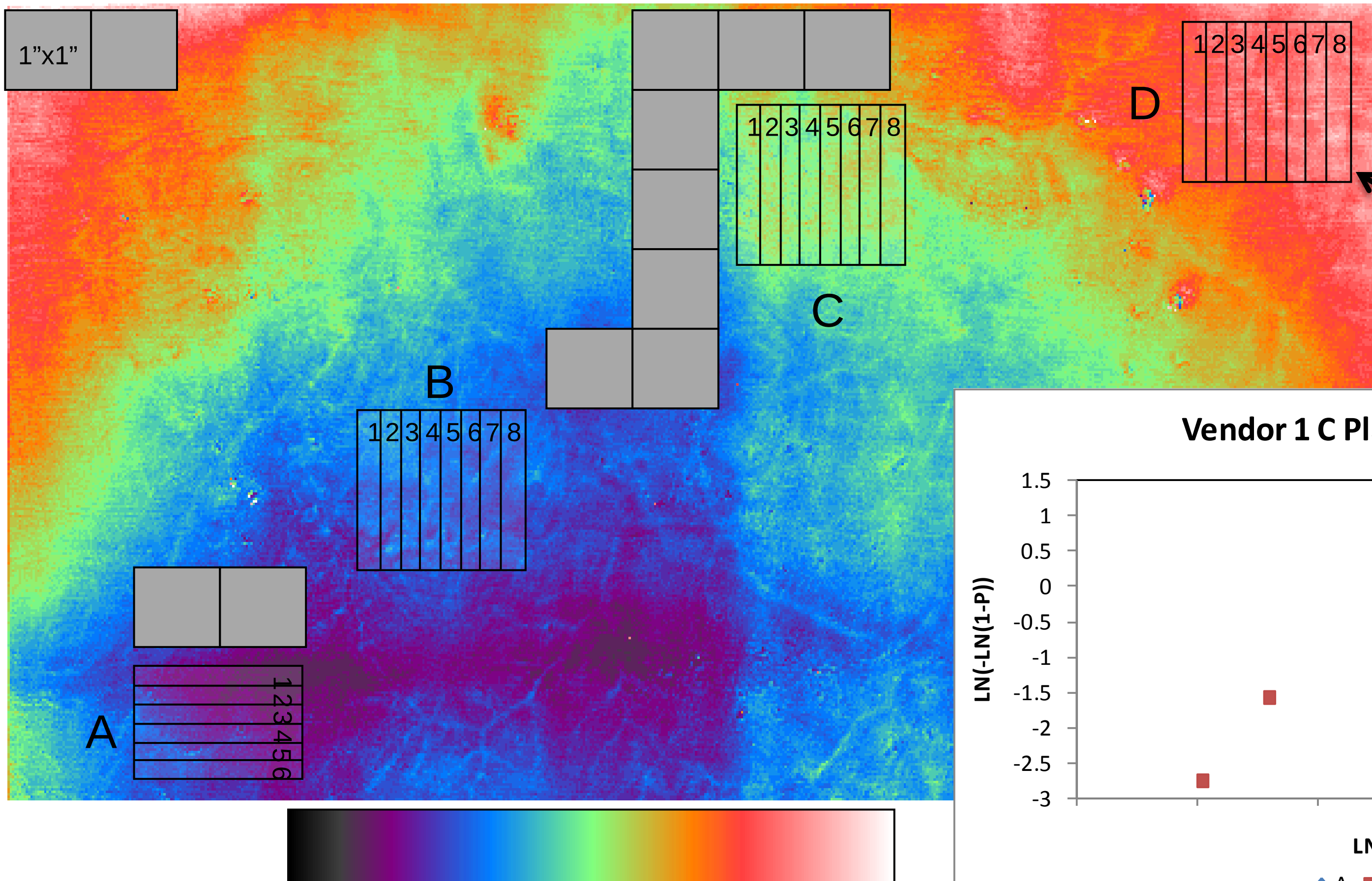
Destructive Testing of NDE Plates

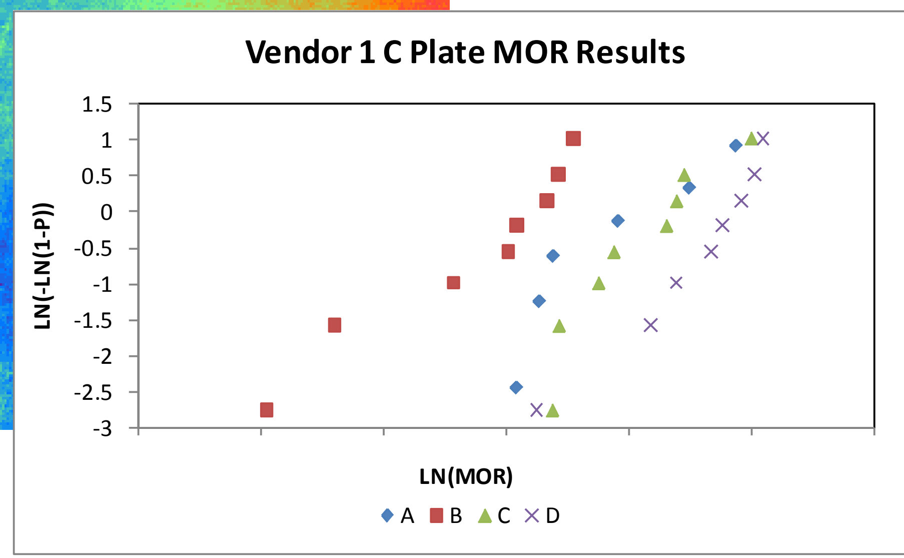
Vendor 1, Plate C



42 Msi

52 Msi

B bar locations



Weibull Plot

Correlation between apparent elastic modulus and strength?

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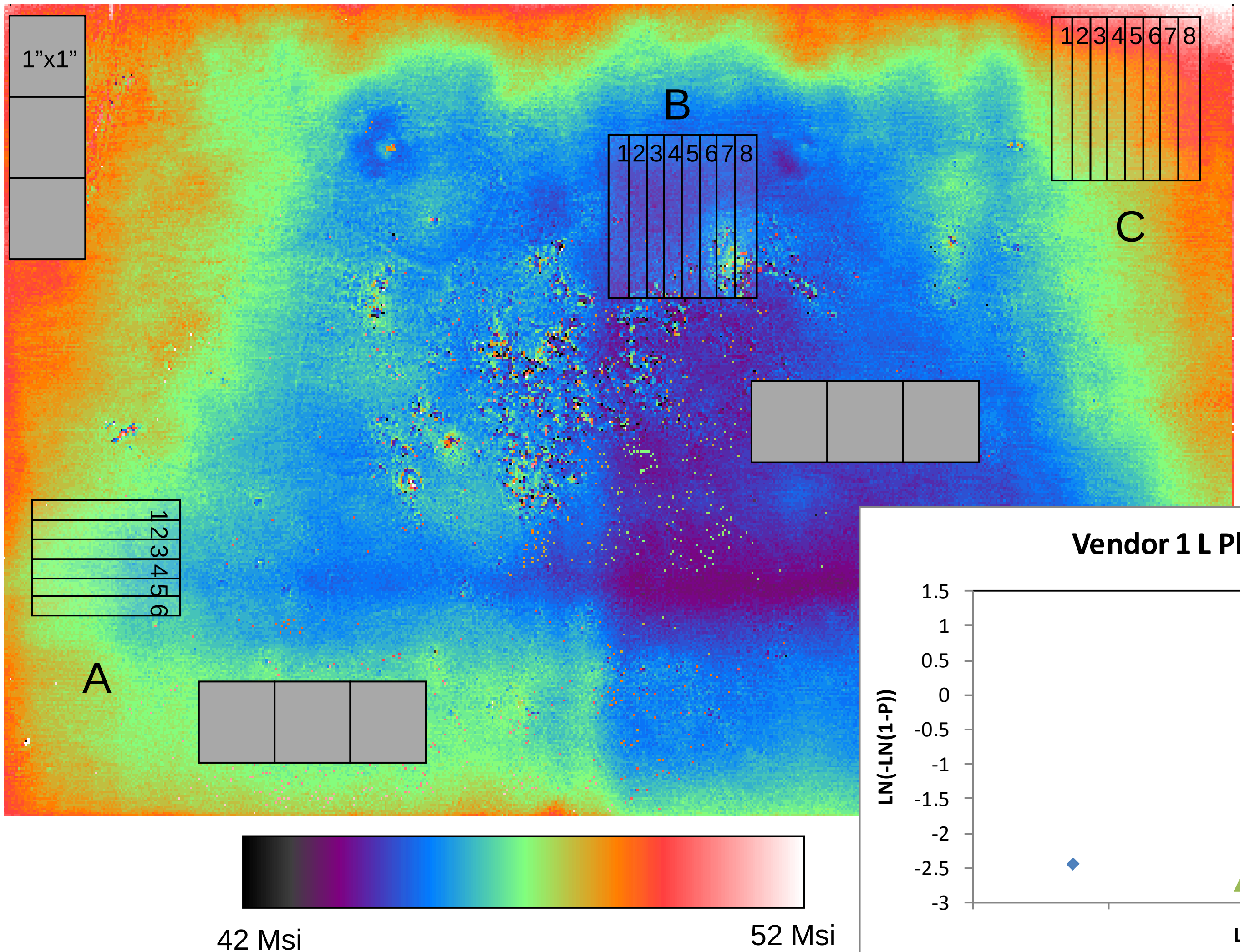
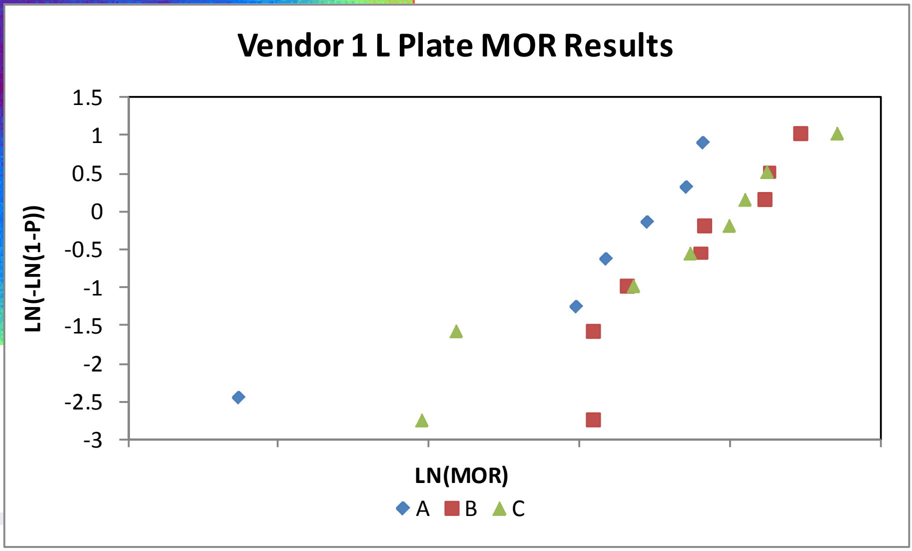
Destructive Testing of NDE Plates

Vendor 1 Plate L





Less correlation between apparent elastic modulus and strength

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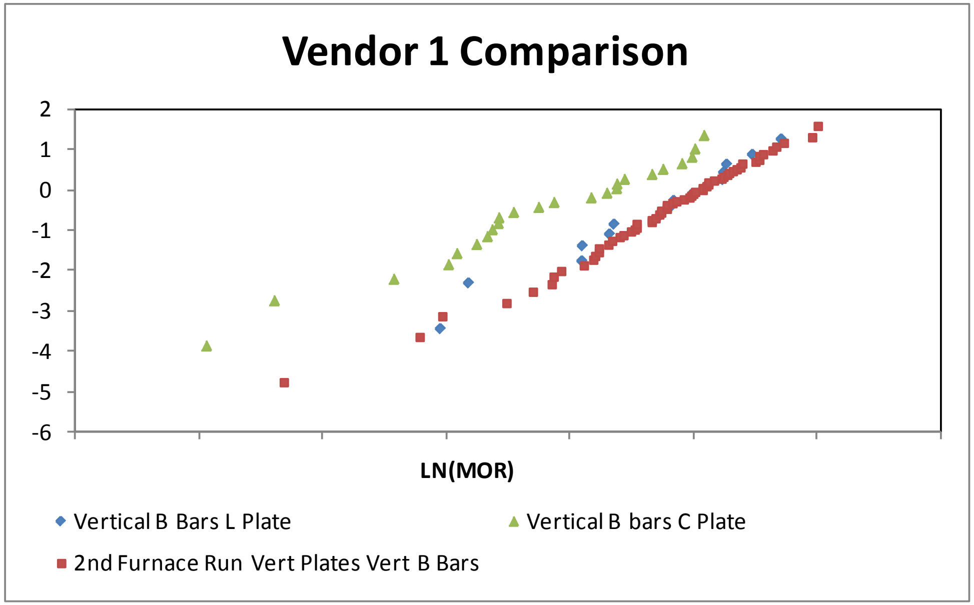
Vendor 1 Results Comparison



Vertical B bars refer to samples with length parallel to short edge of plate; comparison to samples with same orientation from a separate furnace run

- Tests based on Anderson-Darling statistics suggest that the results from the four areas in Plate C cannot be combined into a single data set
  - But if we combine just the vertically oriented B bars the result is a good fit to the Weibull distribution
- Results from Plate C vertical B bars are different from second furnace run, but Plate L B bars are statistically indistinguishable

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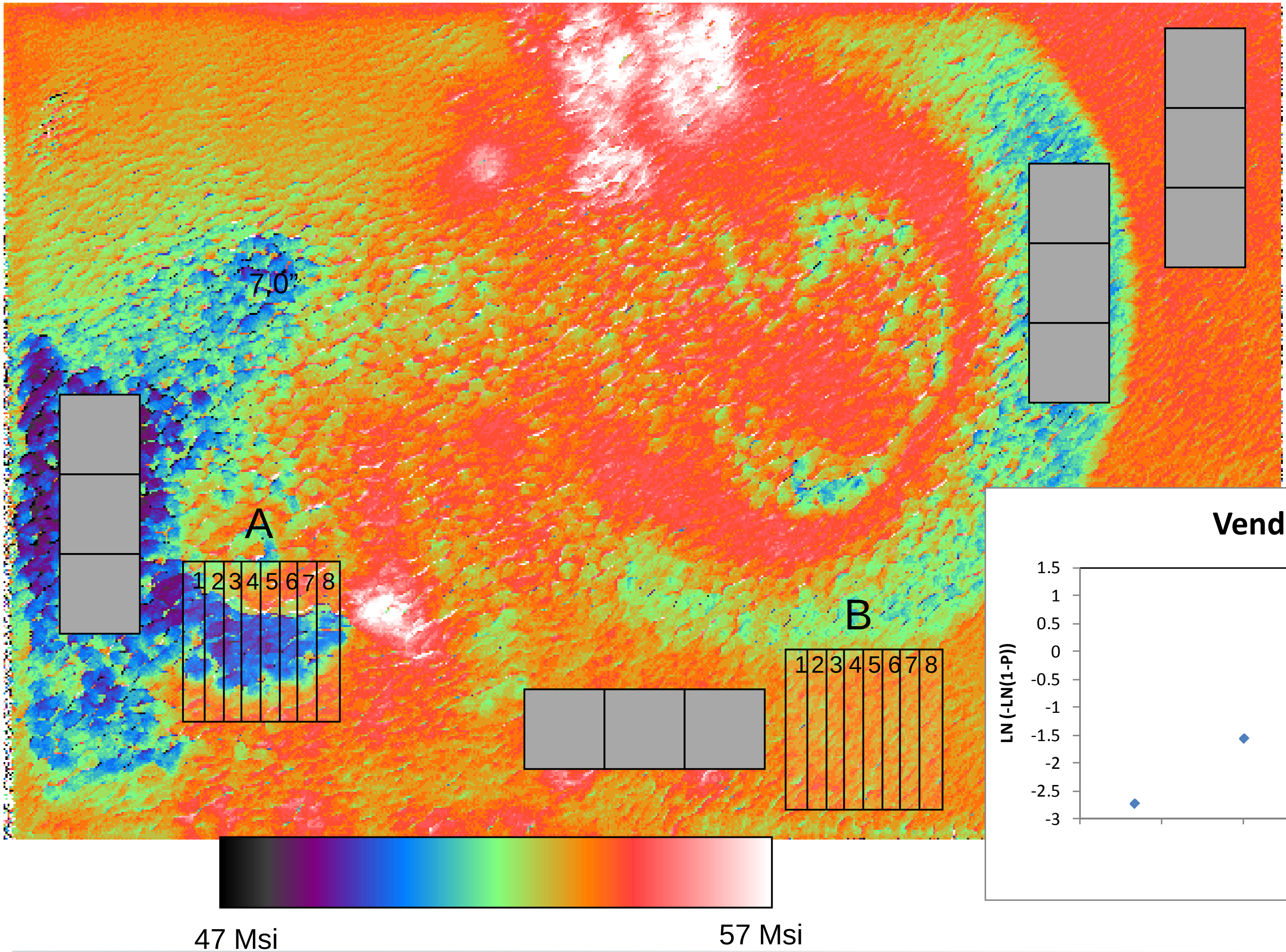
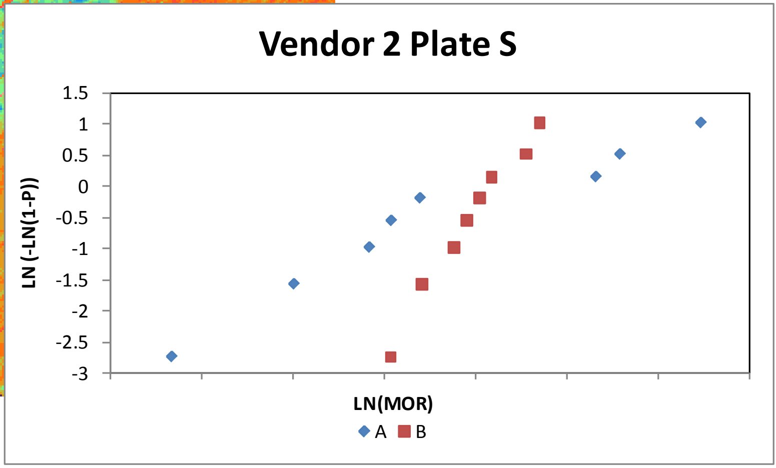
Destructive Testing of NDE Plates

Vendor 2, Plate S





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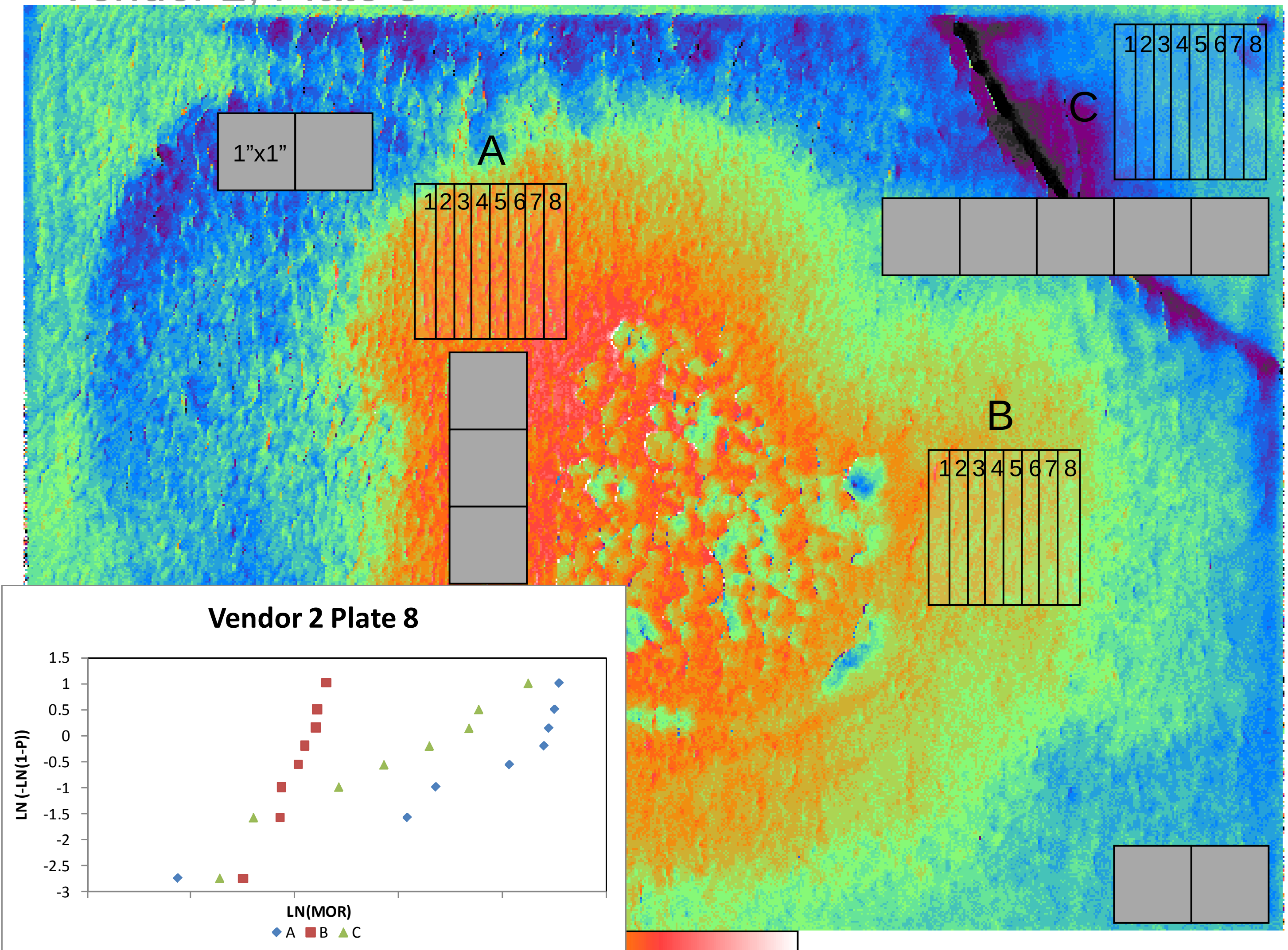
Destructive Testing of NDE Plates

Vendor 2, Plate 8



47 Msi

57 Msi

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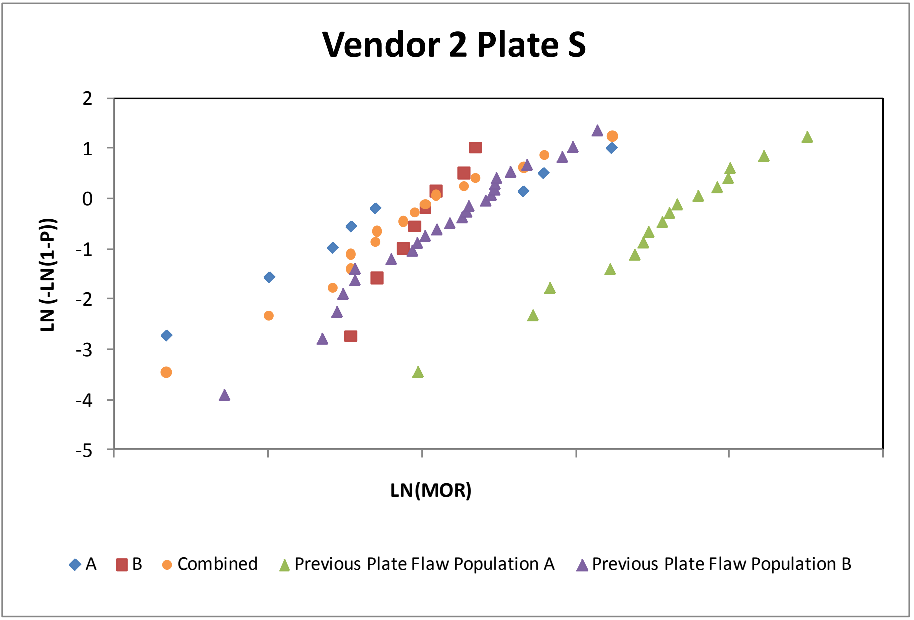
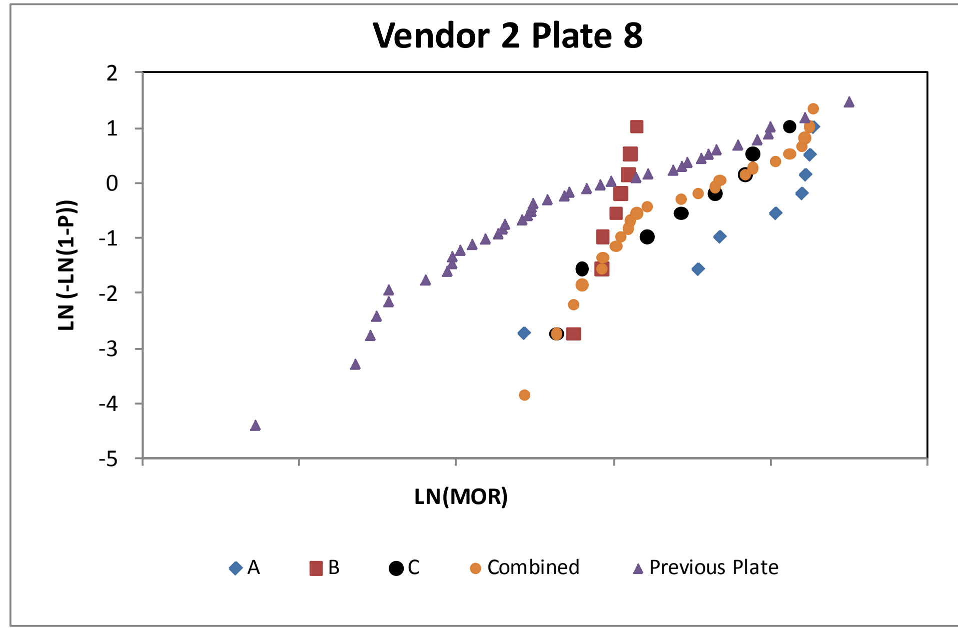
Vendor 2 Results Comparison





- Previous analysis of Vendor 2's material showed that it could be modeled using a partially concurrent flaw model in which one strength controlling flaw type is present in all samples and a second is present in only some samples (Populations A and B in left-hand chart)
  - Locations A and B from Plate S appear consistent with Population B from this previous plate
  - Locations A, B and C from Plate 8 exhibit similar trend to combined data from previous plate

Variations in properties from NDE may be due to relative proportions of co-existing flaw types

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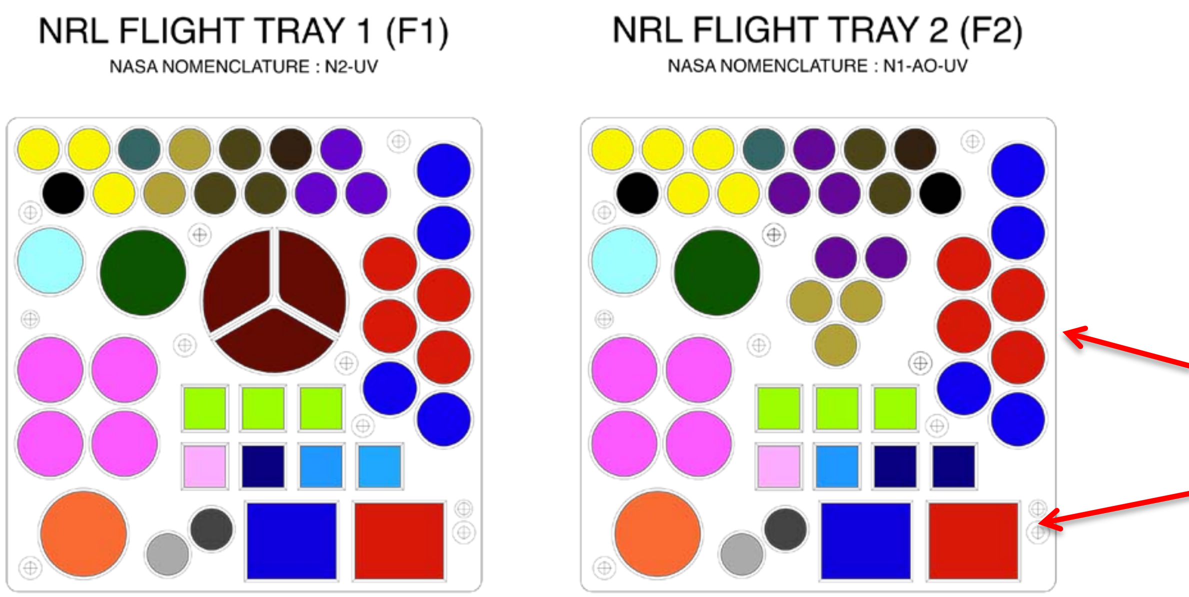
Mechanical Test Specimens on MISSE-6

- Two SiC vendors contributed samples to MISSE-6
  - Both vendors make siliconized SiC (dual-phase Si and SiC) using different methods
  - Both vendors' material has been extensively characterized at Aerospace
- Materials were supplied for two flight trays, traveler trays, and control groups



•Two vendors represented by red and blue

•Circles: EFS disks

•Rectangles: Arrays of 10 closely packed B bars

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MISSE 6 Sample Sources: Vendor 1

- Six 10” x 16” produced in single furnace run
  - *Each plate yielded 42 EFS disks and 46 MOR B bars*
- MISSE-6 samples came from these plates:
  - *20 EFS disks were from a single plate*
    - 4 disks on each flight tray
    - 4 disks on one traveler tray
    - 4 disks labeled traveler but probably never integrated
    - 4 disks in control group
  - *40 B bars taken in equal numbers from two plates*
    - 10 bars on each flight tray
    - 10 bars on one traveler tray
    - 10 bars in control group

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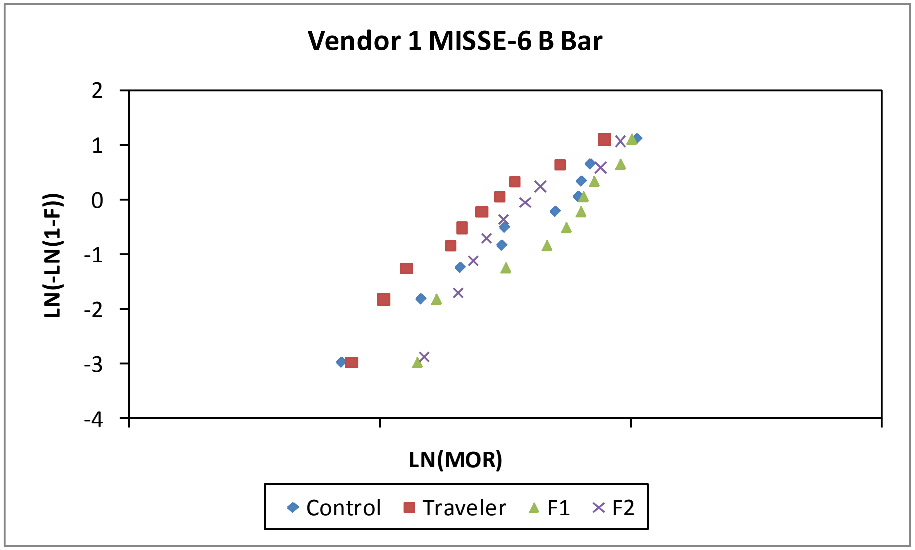
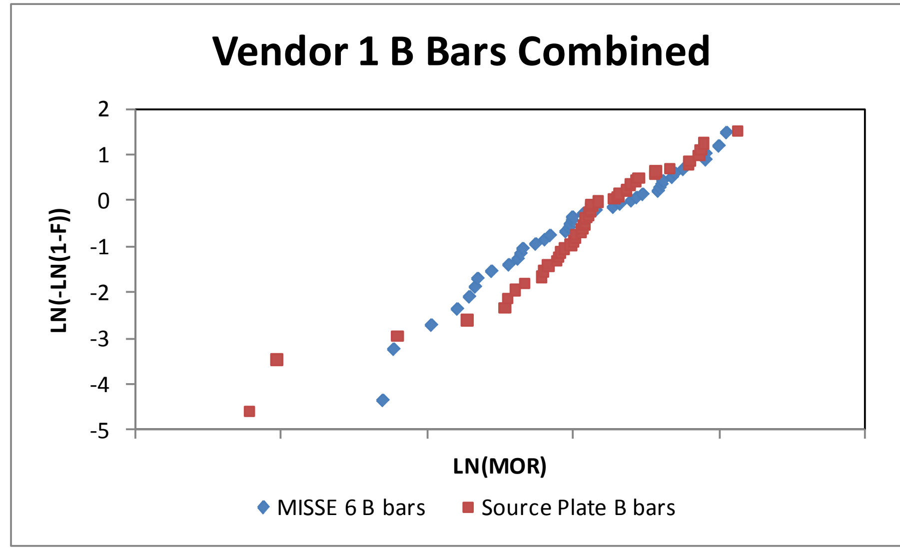
MISSE 6 Vendor 1 MOR Results



MOR results for individual data sets (n = 10)



Results for all vendor 1 MISSE 6 B bars plotted with B bars from source plates

Data sets were compared using multiple statistical methods (all using 5% level of significance):

- Anova and t-tests assume underlying normal distribution; log likelihood assumes underlying Weibull distribution; Anderson-Darling is parameterless
- No significant difference between control, traveler, flight trays, or between MISSE 6 and previous testing of source material

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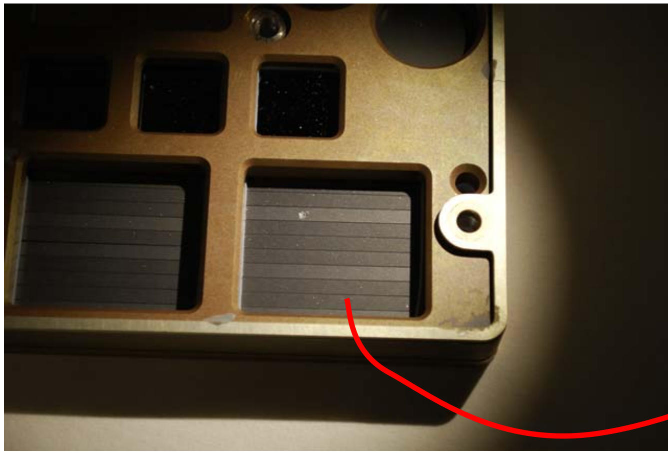
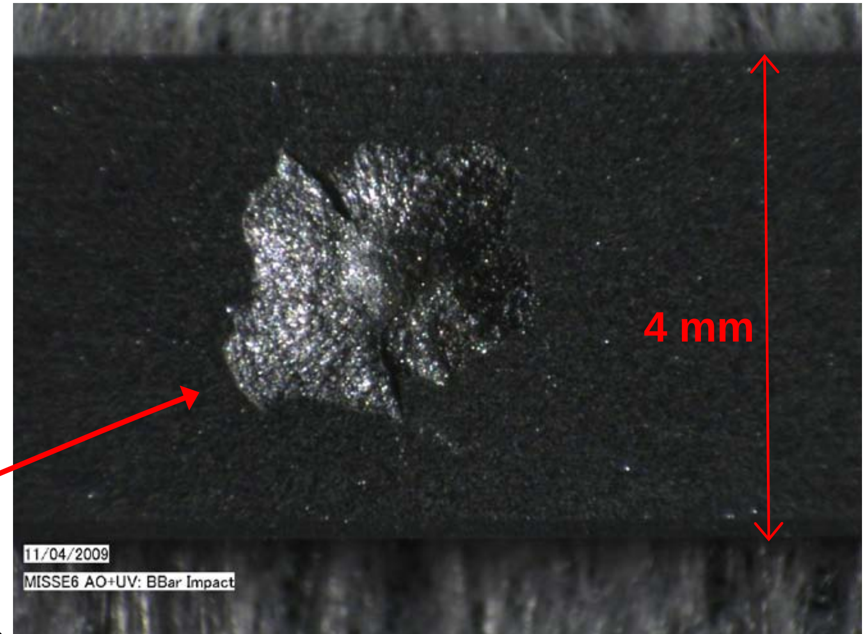
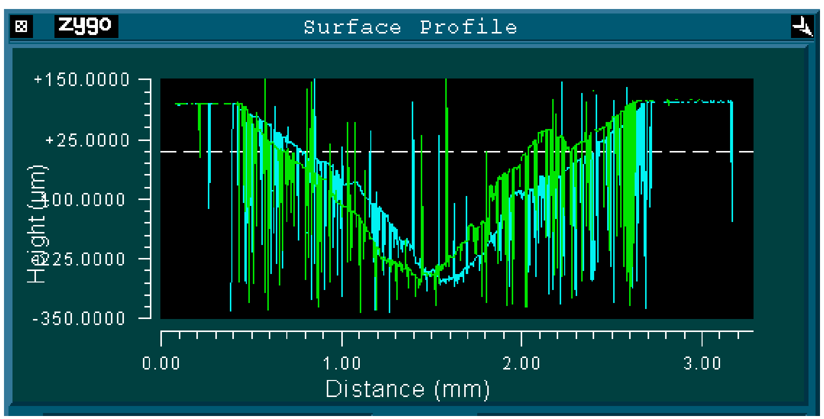
MISSE 6 Vendor 1 B Bar Bonus Feature

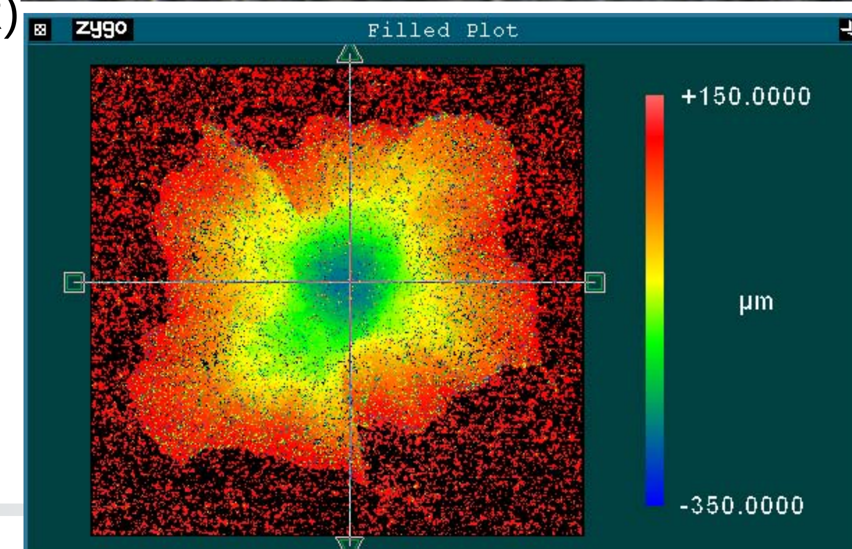
Micrometeoroid Impact





Micrometeoroid impact on B bar, AO/UV tray (B bar not tested for MOR)





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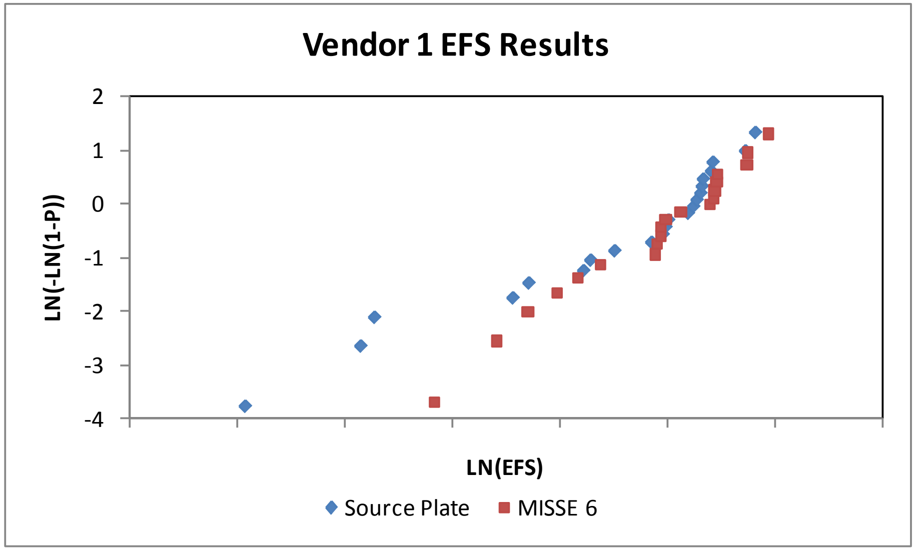
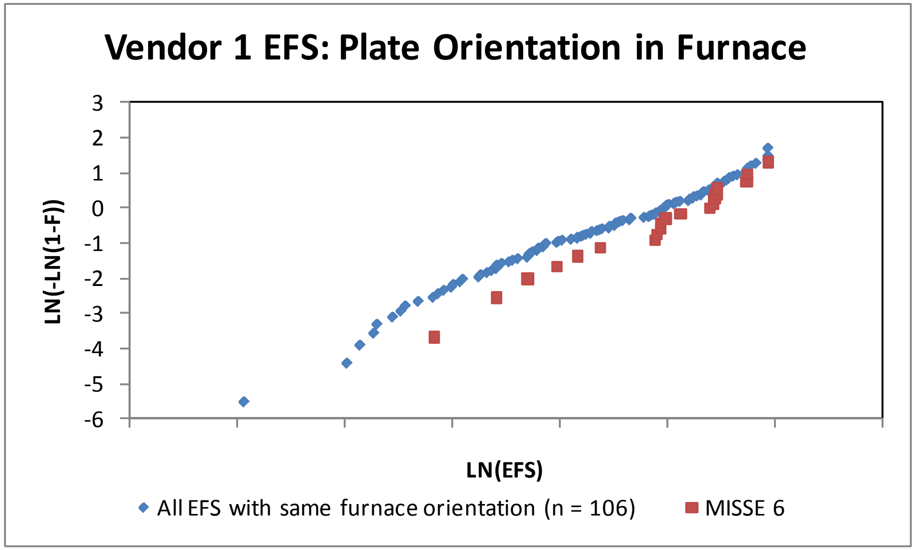
MISSE 6 Vendor 1 EFS Results



Weibull plot of all vendor 1 MISSE 6 EFS results (n = 20) and other EFS results from same source plate (n = 22)



Weibull plot of all vendor 2 MISSE 6 EFS results (n = 20) and other EFS results from source plates with same furnace orientation (n = 106)

- Weibull distribution fits MISSE-6 EFS data but not source material EFS
  - If we calculate Weibull parameters for the source material, log likelihood test shows no signficant difference
- Anova and t-tests assume underlying normal distribution; log likelihood assumes underlying Weibull distribution; Anderson-Darling is parameterless
- No significant difference between control, traveler, flight trays, or between MISSE 6 and previous testing of source material

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MISSE 6 Sample Sources: Vendor 2

- 8” diameter tiles used for EFS disks; 4” square tiles for MOR bars
- MISSE-6 samples came from these plates:
  - *16 EFS disks were from two 8-inch tiles*
    - 4 disks on each flight tray
    - 4 disks on one traveler tray
    - 4 disks in control group
  - *40 B bars were from two 4-inch tiles*
    - 10 bars on each flight tray
    - 10 bars on one traveler tray
    - 10 bars in control group (only 9 were returned)

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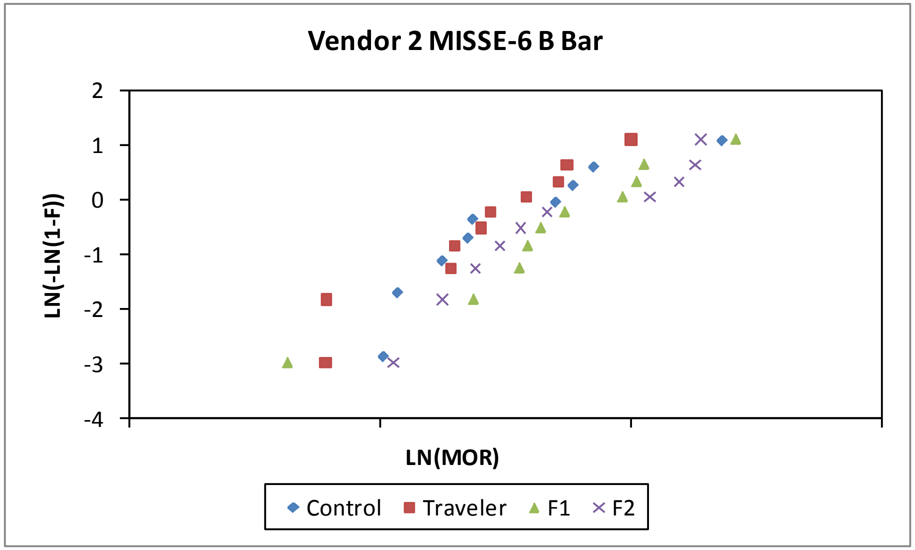
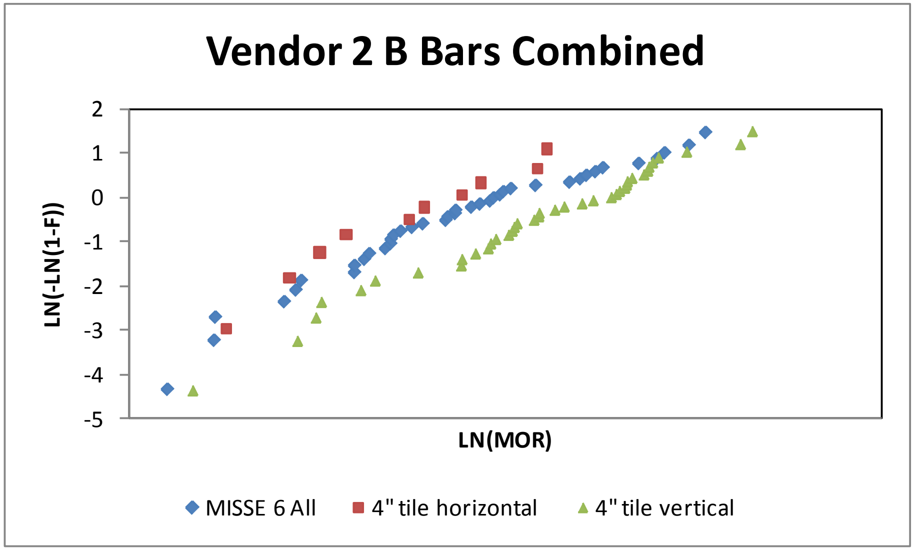
MISSE 6 Vendor 2 MOR Results



MOR results for individual data sets (n = 10)



Results for all vendor 2 MISSE 6 B bars plotted with B bars from similar source tiles

Data sets were compared using multiple statistical methods (all using 5% level of significance):

- Anova and t-tests assume underlying normal distribution; log likelihood assumes underlying Weibull distribution; Anderson-Darling is parameterless
- No significant difference between control, traveler, flight trays, or between MISSE 6 and previous testing of source material

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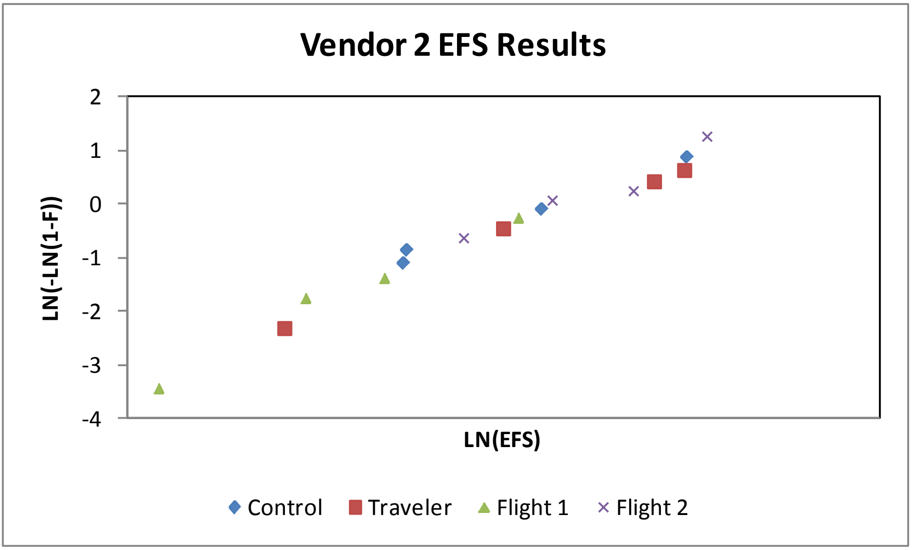
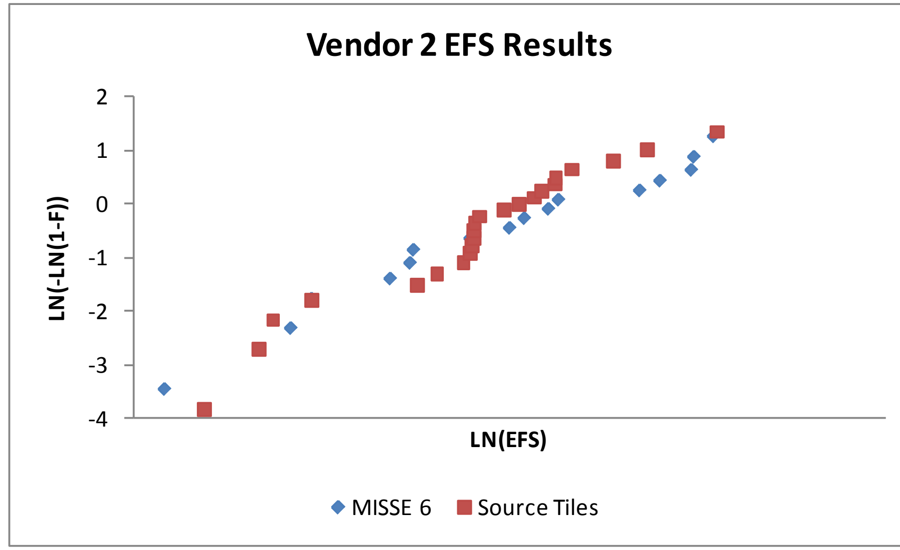
MISSE 6 Vendor 2 EFS Results





All vendor 2 MISSE 6 EFS results (n = 16) in a single Weibull plot

Weibull plot of all vendor 2 MISSE 6 EFS results (n = 20) and other EFS results from source tiles (n = 23)

- Four MISSE 6 sample groups showed no significant differences using Anova or parameter-free comparison
- Combining all four groups gives a single sample (n = 16) that is fit by Weibull distribution
  - No significant difference MISSE 6 samples and previous testing of source material

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Summary

- MOR testing of NDE plates
  - *Vendor 1: Different locations based on modulus variation show differences in strength, but no clear trend*
  - *Vendor 2: Strength controlled by two different flaw populations and elastic property variation measured by acoustic techniques may correlate to spatial distribution of flaws*
  - *NDE techniques illustrated here may have promise for quality inspection at different stages of fabrication*
- MISSE 6 Mechanical testing samples
  - *MISSE 6 does not alter strength of the two kinds of SiC tested:*
    - On-orbit exposure
    - Handling and storage over 3+ years

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